IS 10570 (2011): METHODS OF TESTING REFRACTORY CASTABLES
[MTD 15: Refractories]

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(पहला पुनरीक्षण)

Indian Standard

METHODS OF TESTING REFRACTORY CASTABLES

(First Revision)

ICS 81.080

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

May 2011 **Price Group 3**

Refractories Sectional Committee, MTD 15

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Refractories Sectional Committee had been approved by the Metallurgical Engineering Division Council.

The test methods prescribed in this standard are intended to be used for assessment of the quality of refractory castables as well as for checking their conformity to the standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS OF TESTING REFRACTORY CASTABLES

(First Revision)

1 SCOPE

1.1 This standard prescribes the methods of sampling and tests of refractory castables. It excludes silicon carbide based castables.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1527 : 1972	Methods for chemical analysis of high silica refractory materials (*first revision*)
1528	Methods of sampling and physical tests for refractory materials:
(Part 1) : 2009	Determination of pyrometric cone equivalent (PCE) or softening point (*third revision*)
(Part 4) : 1974	Determination of cold crushing strength (*first revision*)
(Part 5) : 2007/ ISO 5014 : 1997	Method for determination of modulus of rupture at ambient temperature of dense and insulating shaped refractory products (*third revision*)
(Part 7) : 2009	Method of sampling and criteria for conformity (*second revision*)
(Part 12) : 2007/ ISO 5016 : 1997	Method for determination of bulk density and true porosity of shaped insulating refractory products (*second revision*)
(Part 14) : 1974	Sieve analaysis (*first revision*)
(Part 15) : 2007/ ISO 5017 : 1998	Method for determination of bulk density, apparent porosity and true porosity of dense shaped refractory products (*first revision*)
(Part 20) : 1993/ ISO 5103 : 1985	Determination of modulus of rupture at elevated temperature
(Part 23) : 2009	Method of test for dense shaped refractory products — Determination of resistance to abrasion at ambient temperature
4041 : 2006/ ISO 836 : 2001	Terminology for refractories (*first revision*)
4430 : 1979	Specification for mould steels (*first revision*)
12107	Methods of chemical analysis of alumino-silicate refractory materials:
(Part 1) : 1987	Determination of loss on ignition
(Part 2) : 1987	Determination of silica
(Part 3) : 1987	Determination of aluminium
(Part 4) : 1987	Determination of phosphorus
(Part 5) : 1987	Determination of titanium
(Part 6) : 1987	Determination of iron
(Part 7) : 1987	Determination of manganese
(Part 8) : 1987	Determination of calcium and mangesium
(Part 9) : 1987	Determination of sodium and potassium by flame photometry
(Part 10) : 2001	Determination of iron manganese, calcium and magnesium by atomic absorption

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 4041 and the following shall apply.

3.1 Refractory Castables — Mixture of graded refractory aggregates and usually a hydraulically setting cement, primarily of calcium aluminate variety, with no or some addition of other constituent made for specific purposes. The material is usually supplied dry. It may be installed by vibro-casting or ramming with the addition of appropriate amount of water or other liquid, which may serve as binder.

3.2 Classification of Castables

3.2.1 *Conventional* — A castable containing greater than 2.5 percent calcium oxide on calcined basis.

3.2.1.1 *Dense castable*

3.2.1.2 *Insulating castable* — Bulk density having a maximum of 1.85 g/cc (Measured as per **8.3.2** and **8.3.4**).

3.2.2 *Low Cement Castable Type-II* — A castable containing calcium oxide greater than 1.0 percent and maximum of 2.5 percent on a calcined basis.

3.2.3 *Ultra Low Cement Type-III* — A castable containing calcium oxide greater than 0.2 percent and maximum of 1.0 percent on a calcined basis.

3.2.4 *No Cement Castable Type-IV* — A castable containing calcium oxide maximum of 0.2 percent on a calcined basis.

3.2.5 *Gunning Material Type-V*— This material can be in the category of either conventional or low cement depending on its calcium oxide content. The gradation, based on calcium oxide content, is similar to that of castable. The installation of this product, however, is done by gunning machine.

3.2.6 *Precast and Prefired (PCPF) Shapes* — The evaluation of the product shall be done with the specimen made as per this standard, from the same lot.

4 SAMPLING

4.1 Lot

In any consignment, all the containers/bags holding refractory castables of the same type and grade, manufactured by the same firm under similar conditions of production shall be grouped together to constitute a lot. The maximum mass of any lot, however, shall be limited to 50 tonne.

4.2 Sample Size

For completing all the tests, a minimum quantity of about 50 kg is required as sample. As these are normally supplied in 50 kg containers/bags, the number of containers/bags mentioned below shall be selected at random on the following basis:

Lot Size tonne	*No. of 50-kg Containers/Bags to be Selected as Sample*
Up to 10	1
Over 10 up to 25	2
Over 25 up to 50	4

4.3 The selected containers/bags shall be emptied on a suitable dry surface and the material should be thoroughly mixed and reduced to 50 kg by coning and quartering. This final sample shall be divided equally into four test samples by successive coning and quartering. These test samples shall be used for various physical tests such as;

a) Pyrometric cone equivalent (PCE) and sieve analysis;
b) Bulk density and firing shrinkage;
c) Modulus of rupture (MOR) and cold crushing strength (CCS);
d) Thermal conductivity; and
e) Hot modulus of rupture [*see* IS 1528 (Part 20)].

It may be noted that the same test sample may be used for more than one test, if permitted by respective test methods.

4.4 All the test samples tested for various physical and chemical characteristics shall meet the corresponding requirements for acceptance of the lot.

5 SIEVE ANALYSIS

5.1 From the sample set apart for PCE and sieve analysis, 500 g of sample should be taken by the usual procedure of coning and quartering.

5.2 Test Method

Dry sieve analysis of the sample shall be carried out in accordance with IS 1528 (Part 14) using the appropriate sizes of sieves.

6 CHEMICAL ANALYSIS

6.1 Sample for chemical analysis should be selected based on the sieve analysis. After sieve analysis, the different fractions should be ground separately to pass through 212 micron IS sieve. The ground fractions, thus, are recombined in the same proportion and homogenized.

6.2 For alumino-silicate castables, the chemical analysis shall be carried out in accordance with the procedure specified in IS 1527 and IS 12107. Chemical composition of material not covered by these standards shall be done as agreed to between the purchaser and the manufacturer.

NOTE — Alternatively XRF can be used if agreed between the purchaser and the manufacturer.

7 DETERMINATION OF PYROMETRIC CONE EQUIVALENT (PCE)

7.1 Preparation of Test Cones

From the sample set apart for PCE determination, cone and quarter about 50 g. Separate the material on a 150-micron sieve and weigh, grind the two fractions separately to pass through 212-microns IS sieve. The two fractions, thus, ground are recombined in the same proportion and homogenized before preparing the test cones. Cones shall be prepared using the powder sample and adding just enough alkali-free dextrin or other organic binder and water to get a thick paste which may be moulded into cones in the metal mould as specified in IS 1528 (Part 1). The cones

shall be allowed to dry for at least 4 h.

7.2 Test Method

Pyrometric cone equivalent test should be conducted in accordance with IS 1528 (Part 1) using the cones prepared as given in **7.1**.

8 PREPARATION OF TEST SPECIMENS FOR BULK DENSITY, MOR, CCS, LINEAR CHANGE AND THERMAL CONDUCTIVITY

8.1 The size of specimen and the method of preparing test specimen of the material has a marked influence on the indicated properties. Therefore, it is necessary to adopt a standard procedure for preparing the test specimens required for evaluation of the properties particularly bulk density, MOR, CCS, thermal conductivity and linear change.

8.2 Apparatus

8.2.1 *Moulds* — The moulds for casting the various samples should preferably be of steel. A gang mould of 3 or 5 compartments, to fabricate 40 mm × 40 mm × 160 mm specimen, should conform to IS 4430.

8.2.2 *Standard Tamping Rods* — These should be made of non-absorptive seasoned teak wood of cross section 12.5 mm × 25.0 mm and a length of 125 mm to 150 mm. The tamping face shall be flat and at right angle to the length of the tamping rod. A similar rod has been described in IS 4430.

8.2.3 *Planetary Mortar Mixer* — An electrically operated stationary type mixer provided with a rotary mixing blade and multi speed control switch. The quantity of sample for mixing should be so chosen as to occupy at least 40 percent of the bowl volume when mixed. The maximum quantity of dry castable to be loaded in the mixer is such that the dry or wet castable do not get splashed out of the mixer during mixing. This type of mixer is recommended for mixing conventional dense castables but is mandatory for low/ultra low/no cement castables and gunning materials.

8.2.4 *Vibrating Table* — The vibrating table should be flat and horizontal. It should provide only uniaxial vertical vibrations at mains frequency. Preferably the vibration frequency of the vibrating table should be in the range of 3 000-3 600 vpm with an amplitude of 0.8 ± 0.05 mm.

8.3 Preparation of Mix Compaction and Shaping of Specimen

8.3.1 The quantity of sample, as required for each test, is taken from the portion set apart, and is dry mixed thoroughly. Cold water at a temperature not exceeding 20°C should be used for mixing. The quantity of water should be as recommended by the manufacturer of the castable. Alternatively, the quantity of water needed to produce a very stiff ball should be determined by the ball-in-hand consistency test as described in Annex A.

8.3.2 *Hand Mixing of Insulating Castables*

Dry mix 2 kg of castable for 1 min on a non-porous metal plate. Make a cone with a central hollow. Add water in the 'hollow' of the cone and mix with a trowel for 2 min. Soak for 1 min, keeping the material under cover. Mix further for a minute and use it up quickly for making the specimen by tamping/rodding.

8.3.3 *Machine Mixing of Dense Castable*

Requisite quantity of water should be added in the bowl as described in **8.3.1**, then introduce castable sample. The total actual wet mixing time, including water additions, should be ~ 3 min for conventional dense castables, 4 to 6 min for low/ultra low/no cement castables and gunning materials. These do not override any recommendations provided by the manufacturer.

8.3.4 *Compaction of Test Specimen by Tamping/ Rodding*

The compartments of the mould are filled slightly more than half the depth of the mould with the wet castable mix. The mix is then tamped 40 times, the tamping being done along the length of the mould with back and forth movement over the entire face of the mould. While tamping it should be ensured that the aggregates are not greatly damaged. Then the surface is scratched lightly and a further quantity of mix is put in the mould in excess and compact as done for the first layer. The excess material is scrapped off with a metal straightedge using a light sawing action and the surface smoothened by a trowel. This method is adopted only for the preparation of insulating castable samples

8.3.5 *Compaction of Dense Castable by Vibration*

The compartments of the prism moulds are filled with the mix. The material in the moulds is uniformly distributed and vibrated on the vibrating table (*see* **8.2.4**) until the surface become glossy. The height of the overfill should be 5 to 10 mm. The mould is removed from the vibrating table, the overfill frame or collar is removed, the overfill is scrapped off with a metal straightedge using a light sawing action and the surface smoothened with a trowel. The total time for the preparation of mix and for making the test pieces should not exceed 10 min.

8.3.6 Specimens for the gunning mixes are prepared similar to castable as described in **8.3.5**.

8.3.7 Specimens for the PCPF blocks are prepared from the respective dry castables as per **8.3.4** or **8.3.5**, depending on the type of castables.

8.4 Curing of Test Specimen

Subsequent to shaping dense conventional and insulating castables, specimens are cured in 90 percent relative humidity at 18 ± 2°C for 24 h. The relative humidity can be maintained by covering the specimen with wet cloth. The wet cloth, however, should not be in contact with the surface of specimen and it should be ensured that no water drips on the specimen. The low/ultra low/no cement castables can be cured in ambient condition. The curing procedure for the gunning material should be similar to conventional or low cement castables depending on its CaO content. Subsequent to curing, the specimens are dried at 110°C for 24 h. The dried specimens are further heat treated at the temperature for the specified duration as agreed between the supplier and the purchaser for the measurement of various physical and thermal properties.

NOTE — In special cases, the curing schedule as recommended by the manufacturer may be adopted.

9 DETERMINATION OF BULK DENSITY

9.1 Three 40 mm × 40 mm × 160 mm prisms, prepared as described in **7**, shall be tested for determination of bulk density in accordance with IS 1528 (Part 12).

10 DETERMINATION OF APPARENT POROSITY

10.1 Test Specimen

Approximately 40 mm × 40 mm × 40 mm specimens from cured or fired samples, as agreed between the supplier and the purchaser, should be used for the determination of apparent porosity.

NOTE — For the monolithic refractories test specimens which are susceptible to attack by water should be preheated to a temperature so that no part of the refractory body is attacked by water.

10.2 Test Method

Apparent porosity shall be determined according to IS 1528 (Part 15).

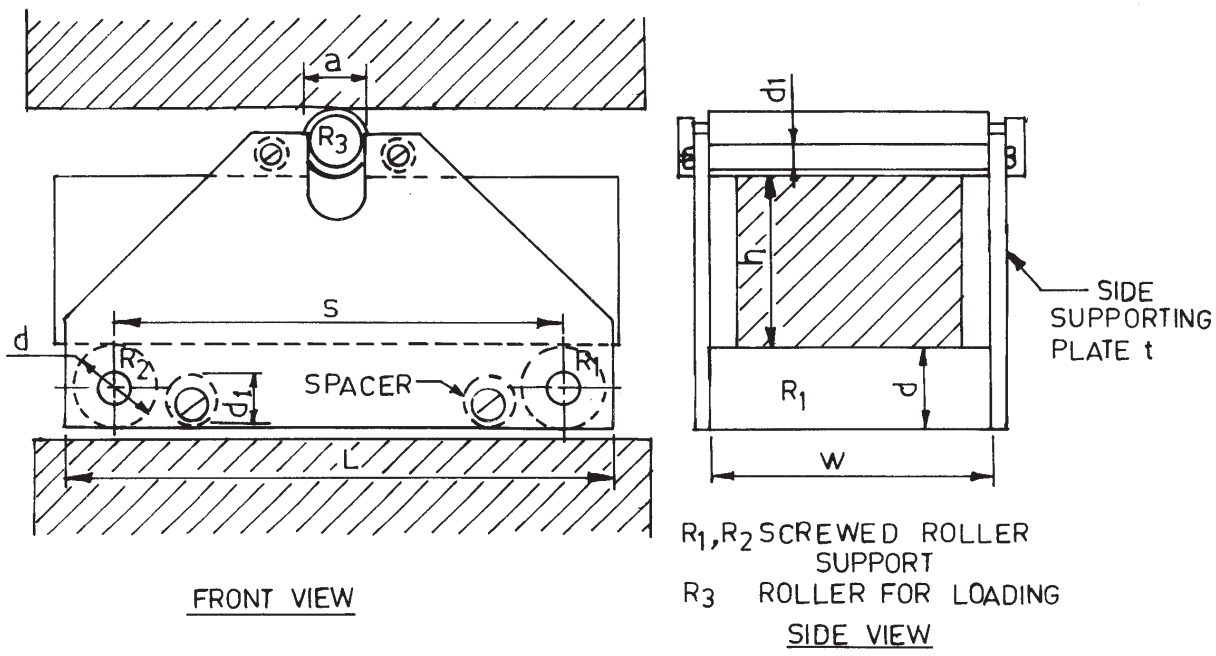
11 DETERMINATION OF MODULUS OF RUPTURE

The cured/fired prisms, used for bulk density test, shall be tested in accordance with IS 1528 (Part 5). The span shall be 10 cm. The individual values of minimum 2 test specimen should be recorded. A convenient jig for loading is shown in Fig. 1.

12 DETERMINATION OF COLD CRUSHING STRENGTH

12.1 Test Specimen

The modulus of rupture measurement yields two numbers of approximately equal sized specimens. These prisms shall be used for determining the CCS. Each half prism shall be tested for compressive strength by applying load on its top face over an area of 40 mm × 40 mm, which shall be placed between two hard metal plates. These plates shall be at least 10 mm thick, 40 ± 0.1 mm wide and 40 ± 0.1 mm long. Their surfaces shall be plane to within 0.02 mm. The



Shape of specimen	*L*	*d*	d_1	*s*	*t*	*a*	*w*	*h*
Prism	220	10	6	100	8	4	50	40

FIG. 1 STEEL JIG FOR MODULUS OF RUPTURE TEST

plates shall preferably be of tungsten carbide or a steel of hardness of at least 600 HV. During the test the two test plates are placed in such a way that the upper plate is maintained vertically above the lower plate with the sample sandwiched between the two. A convenient jig for conducting compressive strength on broken prisms is given in Fig. 2.

12.2 Test Method

Cold crushing strength test shall be determined according to IS 1528 (Part 4). The load shall be applied uniformly. The individual results of minimum four half prisms, generated by modulus of rupture test, shall be reported.

13 DETERMINATION OF LINEAR CHANGE AFTER FIRING

13.1 Test Specimen

Specimens of 40 mm × 40 mm × 160 mm size, that is used for determining bulk density, shall be used.

13.2 Test Method

The prisms, initially cured and dried at 110°C according to **7.3**, are marked at length in 160 mm direction and the distance is measured at the marks. These are set in the furnace ensuring 160 mm × 40 mm face as the base and fired in normal furnace atmosphere to the test temperature with a slow initial rise in temperature, not taking more than 3 h to reach 500°C and thereafter, at the rate of 200°C to 250°C per hour to the desired temperature. The specimens are held at the final test temperature for 3 h. Carbon bearing formulations should preferably be fired in reducing atmosphere to avoid oxidation of carbon. The reducing atmosphere around the specimen can be created by embedding the same in carbonaceous material like pet coke or carbon black during firing. The permissible variation from the desired maximum temperature shall be ± 10°C. After cooling the fired prisms to room temperature, the distance between markings made prior to the firing of specimens shall be measured. The shrinkage or expansion shall be calculated and reported in percentage with respect to the original dimensions. The individual values of linear change of two test specimens for each temperature should be reported.

14 DETERMINATION OF THERMAL CONDUCTIVITY

14.1 The thermal conductivity test shall be carried out by any suitable method as agreed to between the supplier and the purchaser. The specimens prepared for the measurement should be prefired at 800^0C to ensure that the specimen is devoid of any chemically bonded or physically held water.

15 DETERMINATION OF ABRASION RESISTANCE AT ROOM TEMPERATURE

The abrasion test shall be carried out as per the test processes given in IS 1528 (Part 23).

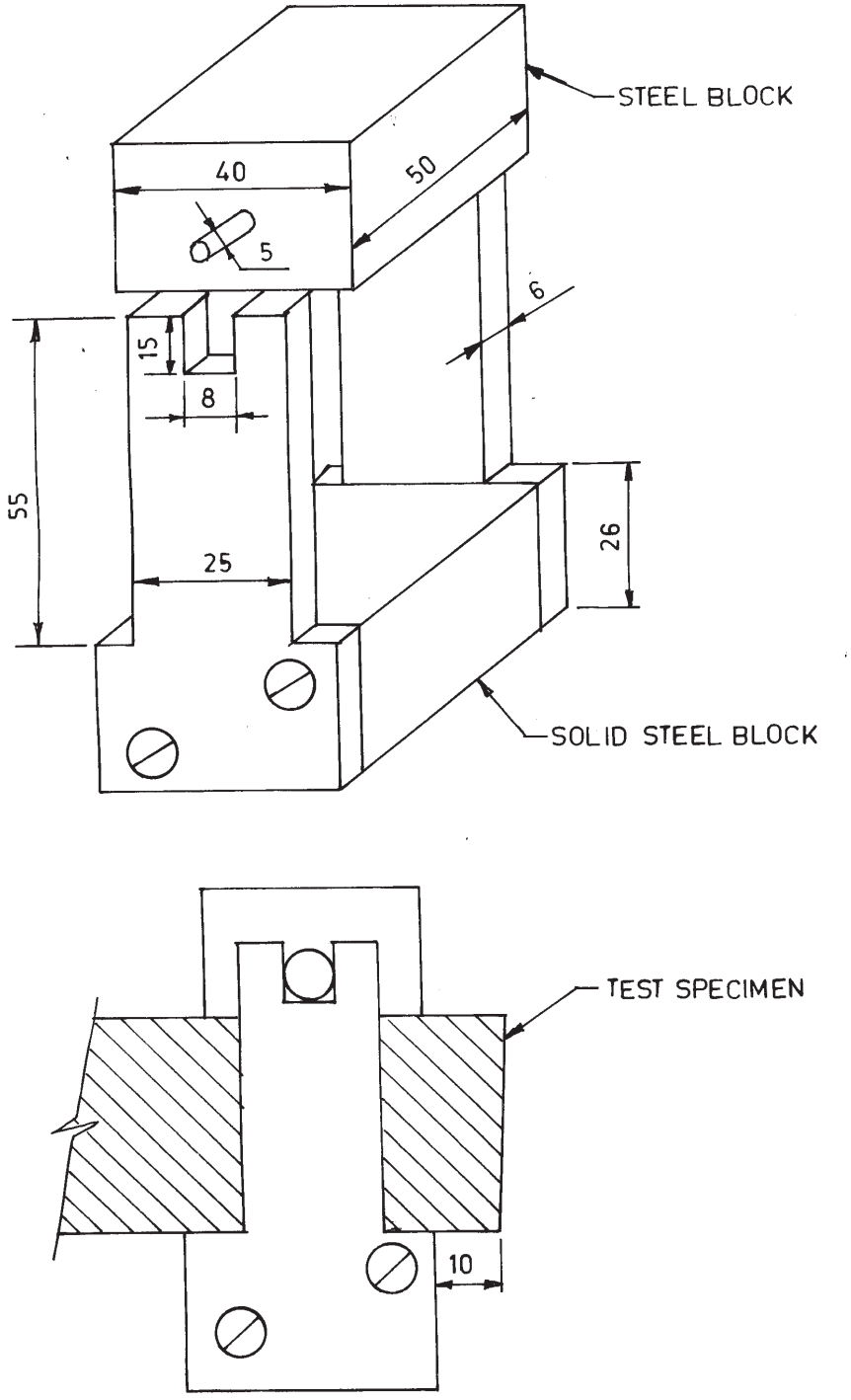
16 DETERMINATION OF HOT MODULUS OF RUPTURE

16.1 Test Specimen

Specimens of 25 mm × 25 mm × 150 mm or 40 mm × 40 mm × 160 mm size shall be used for hot modulus of rupture test.

16.2 Test Method

The prisms initially cured and dried at 110°C shall be used to conduct the test in accordance with IS 1528 (Part 20). The sample may also be prefired as agreed to between the supplier and the purchaser.



All dimensions in millimetres.

FIG. 2 LOADING ARRANGEMENT FOR CCS ON BROKEN PRISMS AFTER MOR TEST

ANNEX A
(*Clause* 8.3.1)

BALL-IN-HAND TEST FOR DETERMINING CONSISTENCY

A-1 The mixed batch shall be of pudding consistency, and the exact amount of water required to produce this condition shall be determined by the trial batch. To determine when the proper consistency has been attained, form a compact ball of the mix in the hand, toss upward about 30 cm and catch it in one hand.

A-2 If material oozes between the fingers it indicates that the water is in excess. On the other hand if ball breaks or shatters it indicates that there is less water than required. The ball should retain its shape when tossed up and held in one hand as it comes down.

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